

Review

Structural Build-Up of Cement Pastes: A Comprehensive Overview and Key Research Directions

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Abstract: The advancement of modern concretes, such as printable concrete, fluid concrete with adapted rheology, and ultra-high-performance concrete, has increased the importance of understanding structural build-up in cement-based materials. This process, which describes the time-dependent evolution of rheological properties, is a key factor to ensure the stability of concrete by influencing segregation, bleeding, formwork pressure, numerical modeling, and multi-layer casting. As a result, the structural build-up of cementitious materials has become a significant area of research in recent years. The structural build-up of cement based-materials results from both a reversible part (thixotropic behavior), driven by colloidal interactions, and an irreversible part, caused by cement hydration and the formation of C-S-H bridges. Various experimental techniques have been developed to investigate these processes, with various factors affecting the thixotropic behavior and overall structural build-up of cement suspensions. This review provides a comprehensive analysis of the current understanding of structural build-up in cement pastes. It covers measurement methods and key influencing factors, including the water-to-binder ratio (w/b), admixtures, temperature, and supplementary cementitious materials (SCMs). By consolidating the existing knowledge and identifying research gaps, this review aims to contribute to the development of sustainable, high-performance cement-based materials suitable for modern construction techniques.

Keywords: structural build-up; thixotropic behavior; cement paste; measurement methods; influencing factors



Received: 6 March 2025

Revised: 25 April 2025

Accepted: 8 May 2025

Published: 13 May 2025

Citation: Hayek, M.; El Bitouri, Y.; Bouarab, K.; Yahia, A. Structural Build-Up of Cement Pastes: A Comprehensive Overview and Key Research Directions. *Constr. Mater.* **2025**, *5*, 31. <https://doi.org/10.3390/constrmater5020031>

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1. Introduction

The construction sector is currently facing various challenges such as cost control, the increasing technicality of works, and climate issues, particularly the carbon footprint. The sector is looking for solutions that make it possible to industrialize and robotize processes, rationalize the use of materials, and reduce construction times and environmental impacts [1,2]. In recent years, a new technology for implementing cementitious materials has therefore been developed. This technology is called “3D printing” in reference to additive manufacturing technologies developed for other materials. This technology encompasses several processes and different techniques for shaping the cementitious material without rigid formwork, which allows for considerable time savings and reduced

labor. The placement via extrusion or multi-layer casting is the common process in the different 3D printing techniques [3,4]. This process consists of extruding the material layer by layer using a numerically controlled nozzle. As the material must be pumped and introduced into the robot, there are rheological requirements on its initial fluidity or what is called “pumpability”, as well as its ability to exit the robot nozzle called “extrudability” [4]. This fluidity must indeed be greater than a threshold value, which depends on the pumping system used (technology and pumping distance, etc.). In addition, to obtain the optimal properties, the different layers placed must structure themselves quickly after placement. This property is often called “buildability”, “printability”, or “structural build-up”, and involves complex physicochemical processes [4–6]. It reflects the time dependence of the rheological behavior of cementitious materials. In fact, when the material is kept at rest, a structural build-up characterized by an increase in the rheological properties occurs.

At the fresh cement paste scale, which can be described as a highly concentrated colloidal suspension, where solid particles are dispersed in a water-based solution [5], the rheological performance is mainly characterized by two rheological parameters, yield stress and viscosity [6]. These parameters are essential for evaluating the fresh state properties of cement-based materials such as flowability, workability, pumpability, and the casting process [7–10]. Over time, due to colloidal interactions between solid particles and the chemical changes, the rheological properties increase with resting time [8,9]. This reflects the structural build-up, which can either be reversible or irreversible. In the reversible case, where the material fully returns to its initial state after a strong re-shearing, the cement paste is classified as thixotropic [11]. A chemically inert material often exhibits fully thixotropic behavior, such as calcium carbonate [12]. In this case, the increase in rheological properties during rest is primarily attributed to particle flocculation. Upon applying shear stress, the calcium carbonate paste completely returns to its dispersed state [12]. However, in the case of cementitious materials, the structural build-up results from both a reversible part, driven by colloidal interactions, and an irreversible part, caused by chemical changes and the formation of hydrates [13–16]. However, it has been observed that the rheological properties of mineral suspensions at rest are influenced by the segregation driven by gravitational forces. In the case of cement-based materials, this influence is observed more in low-concentration cement suspensions (such as cement paste with a w/b ratio of 0.55). In contrast, for highly concentrated cement suspensions (such as those with a w/b ratio below 0.45), this effect is negligible. In fact, the tendency toward segregation becomes negligible when the particles’ volume fraction exceeds the percolation threshold [17–19].

With the apparition of modern concrete such as printable concrete, fluid concrete with adapted rheology (self-compacting concrete), and ultra-high-performance concrete, and as the buildability of concrete materials becomes more important because of its effect on concrete stability (i.e., segregation and bleeding), formwork pressure, and multi-layer casting, the structural build-up of cement-based materials has gained more attention in recent years [2,20–22]. The number of studies on this topic has increased, with various measurement techniques having been developed and numerous influencing factors having been examined. These studies highlight the complexity of the structural build-up mechanisms, the challenges in prediction, the strong interplay between influencing factors, and the significant difficulty in distinguishing between reversible and irreversible processes. However, few studies have focused on the various methods used to assess structural build-up and the interplay of the factors that influence it. Therefore, this review provides an in-depth overview of the current understanding of structural build-up in cement pastes. It provides a detailed discussion about the measurement methods and influencing factors such as the water to binder ratio (w/b), admixtures, temperature, and supplementary cementitious materials (SCMs). By consolidating the existing knowledge and identify-

ing research needs, this work aims to provide valuable insights for both academia and industry, ultimately contributing to the advancement of sustainable and high-performance cement-based materials.

2. Origins and Mechanisms

The structural build-up of cementitious materials is a complex process driven by a combination of physical and chemical effects [5,23]. As shown in Figure 1, a typical time sweep measurement using small-amplitude oscillatory shear shows an increase in both the storage and loss moduli over time, reflecting the structuration of the material. Immediately after mixing (Figure 1a), the cement paste is a suspension of mineral particles dispersed in water. Depending on the flocculation state and the mixing energy, this suspension displays a liquid-like behavior, since the loss modulus is greater than the storage modulus ($G'' > G'$). After a few seconds at rest corresponding to the flocculation characteristic time [24], a percolation continuous path forms due to attractive colloidal forces. This percolation corresponds to the transition point in Figure 1b, where the storage modulus is equal to the loss modulus. Then, a percolated network is formed, and the suspension exhibits solid-like behavior ($G' > G''$) [25,26].

Based on the continuous measurement of the storage modulus, the structural build-up in Portland cement paste can thus be described through at least three main stages [23,26]:

- The first stage corresponds to the non-linear evolution of the storage modulus with time. The mechanisms taking place in this stage are complex. Just after the end of mixing, colloidal interactions between cement particles drive the formation of a network (Figure 1a). According to Han et al. [23], in cementitious pastes characterized by a high ionic strength and a low zeta potential, the attractive Van der Waals forces tend to dominate over the electrostatic forces. This behavior can be attributed to the significant screening effect caused by the high concentration of ions in the solution, which reduces the range and magnitude of the electrostatic interactions between particles. As a result, the attractive Van der Waals forces, which are short-range and non-specific, become the primary drivers of particle interactions. Thus, the attractive Van der Waals forces enable the particles to come together and form a percolated colloidal structure able to withstand minimum stress. This network provides the foundation for the material's subsequent structural evolution. Furthermore, the initial reaction of C_3A can also contribute to the initial increase in the storage modulus. This first stage seems mainly controlled by percolation, notably in cement pastes with a low solid volume fraction or in the presence of admixtures;
- In the second stage, the storage modulus increases linearly with time. The mechanisms behind this increase are a subject of debate and do not achieve a consensus because of the interplay of the roles of particle interaction, cement hydration, and hydration product interaction. One of the most widely accepted hypotheses is that the observed increase in the storage modulus at this stage results from the formation of early hydration products, such as calcium–silicate–hydrate (C-S-H) [25] and ettringite, which create rigid bridges between the cement particles [24]. These hydration products act as mechanical connectors, reinforcing the initial network and transforming it into a more rigid and interconnected framework [27,28];
- The third stage overlaps with the acceleration period of cement hydration and the onset of setting. In this stage marked by an exponential increase in the storage modulus, the inter-particle connections are further strengthened through the continued hydration and densification of the microstructure. The network becomes increasingly rigid as more hydration products fill the pore space. This rigidification [29] is mainly due

to the increase in the contact area between cement particles due to the precipitation of C-S-H.

Recently, by coupling the storage modulus evolution and cumulative heat measured with ex situ isothermal calorimetry in Portland cement paste at different w/b ratios, Michel et al. [30] have shown that structural build-up at rest can be described with two regimes rather than three stages. In fact, they showed that the evolution of the storage modulus as a function of cumulative heat results in a single exponential evolution throughout the induction (second stage) and acceleration periods (third stage). Michel et al. [30] suggest that structural build-up in both periods involves the same mechanism, probably the formation of C-S-H at the contact points between cement particles. However, the first stage was not examined due to experimental complexity. The approach adopted by Michel et al. [30] allows for deconvoluting hydration kinetics from the mechanisms behind the structural build-up. However, this approach does not consider the role that the dissolution of cement particles and supersaturation of the interstitial solution could play in the first and second stages. In fact, the dissolution of cement particles supersaturates the interstitial solution and increases its ionic strength (Figure 1c). This can promote the flocculation of the particles (especially the finest ones) and leads to the precipitation of hydrates (ettringite and some C-S-H [31]) on the surface of the particles. The increase in the storage modulus during the second stage reflects the rigidification of the percolated network (Figure 1c,d) due to the formation of hydrates (probably C-S-H and/or ettringite) at the contact points between cement particles [29,32], but flocculation induced by the dissolution of cement particles can explain in part this increase. This point requires further investigation, since it can explain the reversibility of the structural build-up [11].

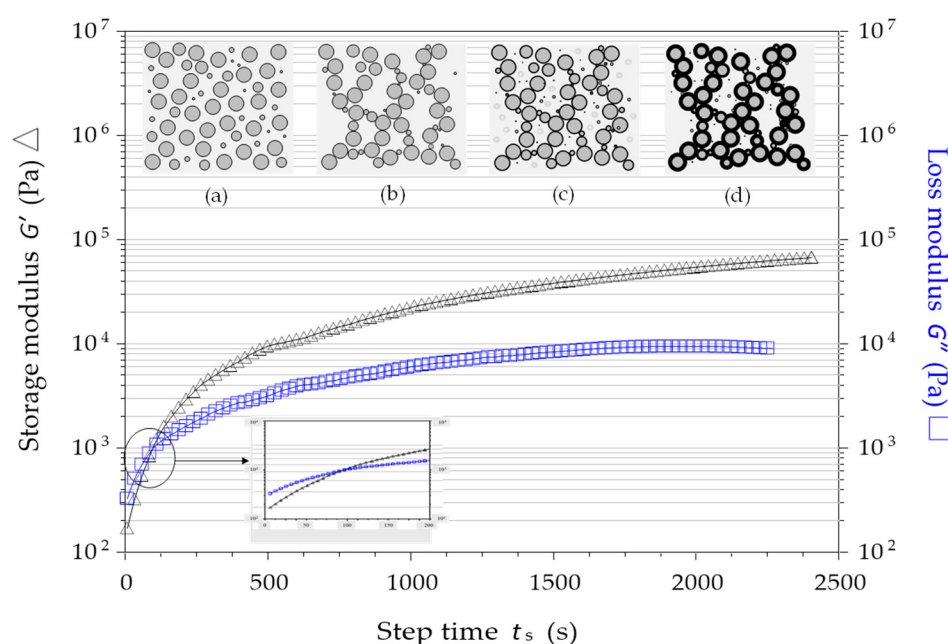


Figure 1. Structural build-up of cement paste (w/b = 0.45) under time sweep measurement. (a) Dispersed state just after the end of mixing. (b) Formation of percolated network. (c) Dissolution and early hydrate formation. (d) Rigidification of percolated network. This figure is based on literature theories and assumptions related to the mechanisms of the structural build-up [24,33,34].

3. Measurement Methods

Different rheological approaches can be used to characterize the structural build-up of cement-based materials. Each approach has its advantages and possibly its limitations. The first approach that was used to describe thixotropy/structural build-up of cement-based

materials is referred to as hysteresis loop. This method has several limitations, as reported by Banfill [35]. The most relevant approach consists of the evolution of the storage modulus over time under small-amplitude oscillatory shearing, since it allows for following the structuration without disturbing the sample during the test [36]. This method may, in some cases, have limitations. Another practical method is based on the determination of the evolution of static yield stress with the resting time, but this method is destructive. However, it makes it easier to relate the static yield stress to practical measurements, such as the mini-slump test or Vicat needle test. Other rheological approaches exist, such as creep tests or stepwise shear.

3.1. Hysteresis Loop Test

A hysteresis loop experiment, as shown in Figure 2, consists of determining the upward and downward flow curves of the material subjected to an increasing and decreasing shear rate (or shear stress). This evolution is carried out either continuously or in stages by applying a constant shear rate for a certain duration. The upward curve reflects the continuous breakdown of the flocculated structure of fresh cement paste subjected to shear stress, while the downward curve describes the process of structure reformation.

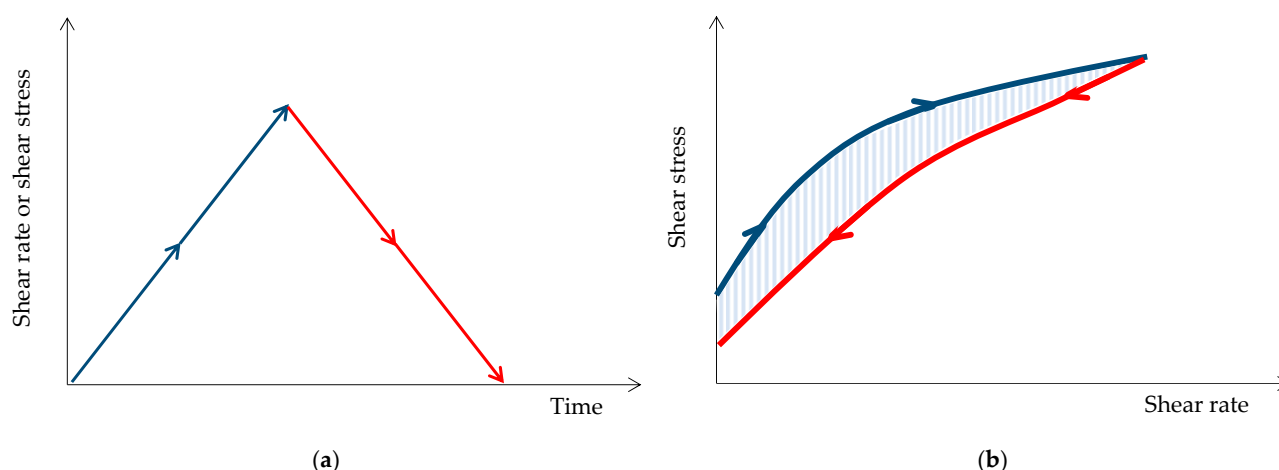


Figure 2. Hysteresis loop test: (a) imposed shear rate (or stress) and (b) response to ascendant (blue) and descendant shear rate (red) ramp.

The variation in the enclosed area between the downcurve and the upcurve over time (hysteresis loop cycles) is an indicator of the structuration of the material. However, as reported by Banfill [35], the hysteresis loop only proves the structural breakdown that occurred during the test, and an infinite number of different loops are possible depending on the experimental conditions [37]. In fact, the shape of the hysteresis loop is directly related to the experimental duration [37,38]. As pointed out by Roussel [38], if the shear rate is applied for a duration less than the flocculation characteristic time, the steady state is not reached. In this case, the measured shear stress curve depends on the flocculation state of the sample. So, if the flocculation in the sample is greater, the steady state cannot be reached, i.e., the deflocculation process (downcurve) does not have enough time to induce a steady state breakdown of the structure, while if the flocculation in the sample is less than the deflocculation equilibrium state, the rebuilding process does not have enough time to bring the structure to its equilibrium state [38]. It is worth noting that the equilibrium time to reach the steady state depends on the shear rate levels. At a high shear rate, the steady state is reached more quickly.

As a preliminary approach, the hysteresis loop test can be performed to assess the structural breakdown and rebuilding of fresh cement paste, but it is important to note that the results are highly dependent on the test conditions.

3.2. Static Yield Stress Measurements

Static yield stress reflects the minimum shear stress required to initiate the flow, i.e., the shear stress corresponding to the flow onset [39–46]. It originates from attractive colloidal forces and direct contact between particles, which tend to form a percolated network of interacting particles able to withstand the minimum stress [39]. It is generally measured through the stress growth procedure with Vane geometry [47]. The suspension is first subjected to a strong pre-shear phase followed by a resting time. Then, a very low and constant shear rate in the range of 10^{-2} – 10^{-3} s^{-1} (depending on rheometer sensitivity) is applied. The evolution of shear stress with time (or strain) is recorded and an illustration of the typical curves is shown in Figure 3, where the peak in shear stress (Figure 3) corresponds to the static yield stress (value of the peak). In some cases, the shear stress evolution does not display a peak, notably in the case of fluid suspension (such as cement suspension with high w/b ratio of 0.55), or in the case of a low resting time (Figure 3) [48]. In these cases, only a plateau corresponding to the steady state can be observed (Figure 3). It should be noted that the static yield stress or the steady state are reached after a few seconds when the shear strain is about 5–15% (Figure 3b). This time corresponds to the duration required to break down the structure of the suspension, and the corresponding strain reflects significant changes in the network of interacting particles that lead to the onset of flow [49]. Roussel et al. [24] and Fourmentin et al. [50] reported that the shear stress–shear strain curve can exhibit an abrupt slope change in the very first stages of the shearing process (when the shear strain is lower than 0.4%). This slope change associated with a very small strain characterizes a rigid network.

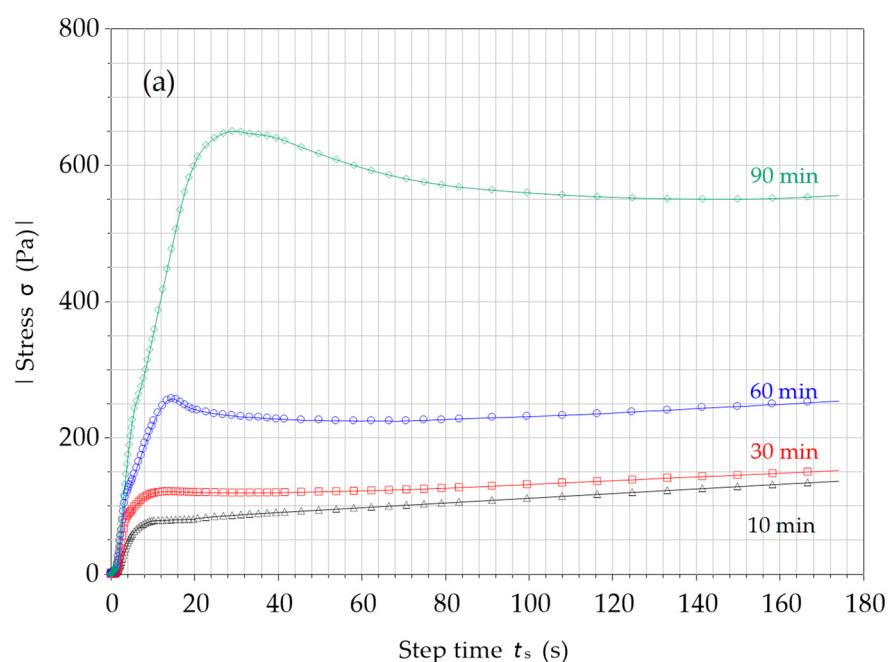


Figure 3. Cont.

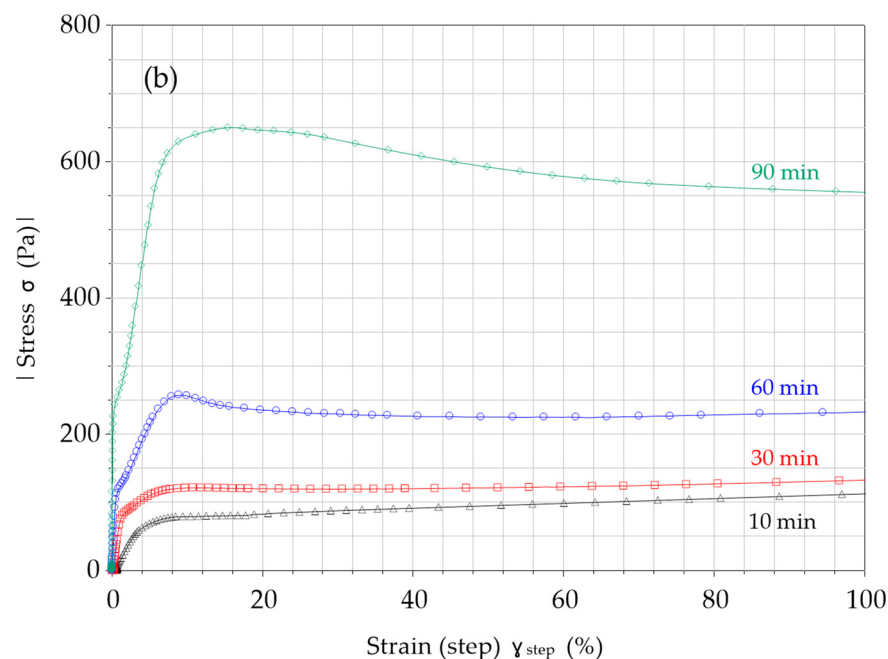


Figure 3. Typical curve evolution of shear stress under low and constant shear rate of 0.005 s^{-1} after different resting times (10, 30, 60, and 90 min). (a) Shear stress vs. time evolution and (b) shear stress vs. shear strain.

Structural build-up can be examined by measuring the static yield stress after various resting times [51–53]. It has been reported that structural build-up and the evolution of static yield stress over time are linked and can be described by different stages. As reported by Zhang et al. [52], the static yield stress increases rapidly during the first few minutes, followed by an induction period during which the static yield stress increases slowly with time. After 60 min of rest, an exponential increase in the static yield stress can be observed (Figure 3 gives an example of this evolution over time). According to Zhang et al. [52], the increase in static yield stress is primarily driven by inter-particle forces, which result from a combination of colloidal interactions and the growing connectivity between C-S-H particles. During the early stage (up to approximately 60 min), colloidal forces play a dominant role in the structural build-up. Beyond this period, the formation and growth of C-S-H phases and other hydration products become the main contributors, progressively reinforcing the microstructure and governing the continued evolution of the static yield stress.

Therefore, the evolution of static yield stress with resting time provides a practical approach to quantify the structural build-up of fresh cement paste. The results can be correlated with practical tests such as the mini-slump test to obtain a more in-depth analysis [54,55]. In addition, by performing a re-shear phase supposed to erase the reversible part of the structural build-up, it is possible to examine the conditions under which the cement pastes exhibit thixotropic behavior [11,56]. However, it should be noted that this test is destructive and does not allow for the continuous monitoring of the structural build-up.

3.3. Small-Amplitude Oscillatory Shearing (SAOS)

SAOS is a non-destructive method for monitoring the structural build-up of fresh cement paste over time [25]. The structural build-up is assessed using a time sweep test through the evolution of the storage modulus (G' , elasticity) and the phase angle over time [36,57]. To ensure that the time sweep test remains non-destructive, the applied oscillatory shear should be below the critical shear strain (Figure 4). Then, it is necessary to determine the critical shear strain and the linear viscoelastic domain (LVED) using a Strain sweep test under an oscillatory strain ranging from 0.0001% to 100% and a constant

frequency (1 to 2 Hz) [58] (Figure 4). The Strain sweep test must begin with a well-dispersed and non-agglomerated cement suspension. To ensure this initial state, a pre-shear is applied at the beginning of the procedure, typically using a shear rate between 100 and 150 s⁻¹ for 30 s (the pre-shear rate and duration should be adjusted according to the specific characteristics of the cement suspension, such as the w/b ratio and the presence of admixtures). This step helps to homogenize the paste and eliminate any initial structuration or particle flocculation.

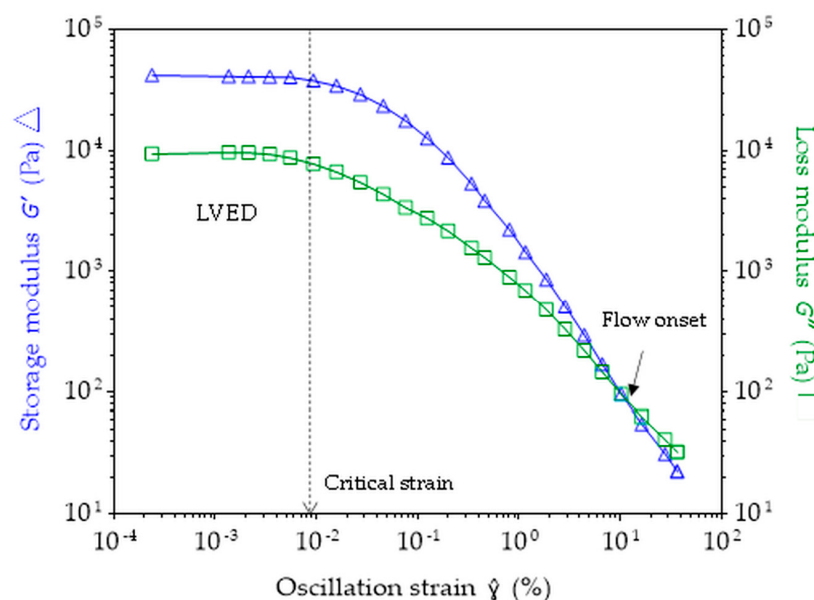


Figure 4. Typical response during a Strain sweep test under a frequency of 1 Hz.

In the LVED, the structure of the suspension is maintained, and the storage (G' , elasticity) and loss (G'' , viscosity) moduli remain constant. The critical shear strain marks the end of the LVED, beyond which the material's internal structure starts to break down. This structural destruction (structural rearrangement or damage, such as the breakdown of flocculated networks) is characterized by a drop in the G' and G'' (Figure 4) [24,49,59,60]. As the oscillatory amplitude strain continues to increase beyond the critical shear strain, the G' and G'' continue to decrease, and the suspension reaches a yielding point (flow onset) when the G' becomes equal to the G'' , and then, the suspension exhibits a liquid-like behavior [61].

Furthermore, under small-amplitude oscillatory shear, i.e., in the viscoelastic domain under a strain amplitude less than that of the critical strain, a time sweep measurement allows for the assessment of the structural build-up over time [36,57] (Figure 5). Mostafa and Yahia [36] suggested to use the under time sweep test, the percolation time, and the rigidification rate to quantify the structural build-up. The percolation time corresponds to the time required for the phase angle to reach its lowest and steady value. This phase angle describes the dephasing between the shear stress and strain oscillation. It varies from 0°, which describes a solid-like behavior where there is no lag between the applied oscillatory strain and the shear stress response, to 90°, which indicates a liquid-like behavior. According to Mostafa and Yahia [36], the percolation time refers to the moment when a stable, interconnected network is first established within the cement suspension. Beyond this point (percolation time), the linear increase in the storage modulus can describe the rigidification rate of the percolated network.

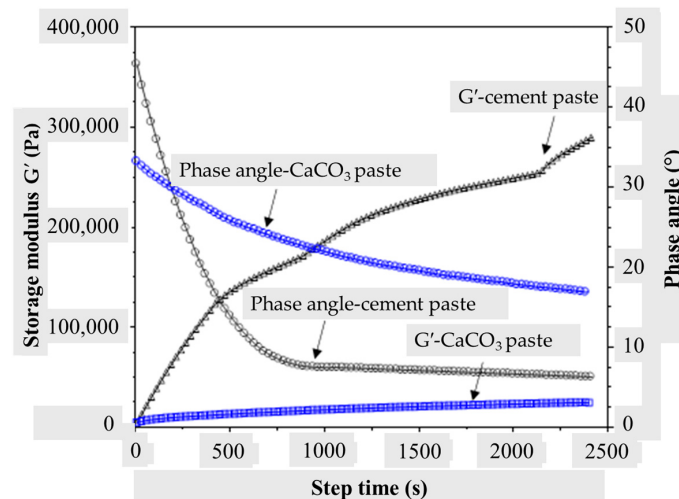


Figure 5. Evolution of the storage modulus and phase angle of cement and calcite pastes ($w/b = 0.50$) under a frequency of 1 Hz.

This type of procedure is the most relevant to examine the structural build-up without disturbing the sample during testing. In addition, this allows for the continuous measurement of the structural build-up, capturing the effect of hydrate formation [30]. However, it requires starting the measurement in the most dispersed state possible to observe the initial increase in the storage modulus. In fact, this initial increase corresponds to the initial flocculation, and the time required to form a percolated path in the suspension is only about few seconds [24]. This time corresponds to the moment when the storage and loss moduli intersect (Figure 6). For instance, it can be difficult to obtain a dispersed state in a very highly concentrated suspension, even with strong mixing and pre-shearing phases. Such a suspension always displays solid-like behavior ($G' > G''$), as shown in Figure 6. This can be attributed to the disruptive shear technique used to obtain the dispersed state of cement paste. As reported by Mostafa and Yahia [62], the oscillatory shear has a more significant effect on dispersing concentrated cement suspensions than the rotational shear. Therefore, an effective approach to enhance the dispersion of concentrated cement paste is the combination of rotational and oscillatory shear, particularly when applying a high angular frequency of 100 rad/s. However, this step introduces complexity, which may restrict the applicability of this method for investigating the structural build-up of cement paste.

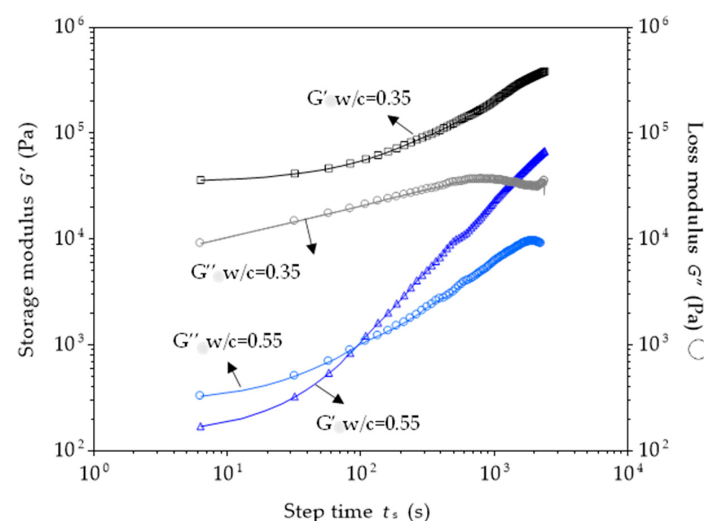


Figure 6. Evolution of the storage and loss moduli with time for cement paste after a strong pre-shear phase.

4. Structural Build-Up Influencing Factors

4.1. Solid Volume Fraction (Or Water-to-Binder Ratio)

The structural build-up of cement pastes is strongly affected by the solid fraction, which includes cement and any mineral additions, or by changes in the water-to-binder (w/b) ratio [63]. An increase in the solid fraction or a decrease in the w/b ratio leads to a higher-volume concentration of particles. This denser packing of particles enhances the number of contact points between them, facilitating the formation of a three-dimensional network. Within this network, colloidal forces become increasingly significant, contributing to a significant increase in both the cohesion of the mixture and the degree of flocculation. These effects promote the development of a more structured and interconnected network, which is a key factor in the structural build-up of cement pastes. Then, denser mixtures exhibit faster structuration due to reduced inter-particle distances. For example, Mostafa and Yahia [36] investigated the effect of the w/b ratio on the structural build-up of cement paste (Portland cement). Their study revealed a significant correlation between a decrease in the w/b ratio and an increase in the build-up rate of the paste. A comparative analysis of cement pastes prepared with w/b ratios of 0.35, 0.40, and 0.45 showed that lowering the w/b ratio enhances the structural build-up kinetics. Similar findings were reported by Kwasny et al. [64], where the structural build-up increased as the w/b ratio decreased in Portland cement pastes prepared with w/b ratios of 0.35, 0.38, 0.40, 0.45, and 0.50. This behavior is attributed to the higher solid volume fraction at lower w/b ratios, which promotes the formation of a denser and more compact particle network. As the distance between particles decreases, inter-particle interactions become stronger, thereby enhancing the structural build-up.

However, studies have shown that variations in the w/b ratio have a limited impact on the nucleation and growth of hydration products, which are primarily responsible for the structural build-up after the percolation process [65]. These findings suggest that the effect of w/b on the build-up of cement pastes is not predominantly governed by the rate or extent of hydration product formation. Instead, it is largely attributed to the intensification and strengthening of the colloidal interactions between particles during the percolation process. In agreement with this observation, Ma et al. demonstrated that a reduction in the w/b ratio primarily affects the strength of colloidal interactions between cement particles, rather than the strength of C-S-H bonds [66].

On the other hand, the effect of the w/b ratio on the rheological properties and structural build-up of cement-based materials cannot be isolated from other factors such as cement fineness, SCMs, temperature, and chemical admixtures. Firstly, studies have shown that the effect of the w/b ratio on the rheological properties of ordinary Portland cement is relatively simple. It is characterized by a decrease in yield stress, viscosity, and build-up rate, driven by enhanced particle separation and reduced inter-particle friction as the w/b ratio increases [64,67]. However, when SCMs are used, this relationship becomes more complex, as variations in rheological properties are also influenced by SCM properties such as water demand, morphology, structure, and reactivity [68–70]. Similarly, water demand, workability, particle interactions, and frictions are also influenced by cement fineness and the presence of chemical admixtures, such as superplasticizers and viscosity-modifying admixtures [71–73]. This effect further complicates the relationship between the variation in the w/b ratio and structural build-up. Therefore, to optimize the buildability of modern concrete mix designs, such as printable concrete and fluid concrete with adapted rheology, it is crucial to understand the variation in rheological properties and structural build-up across different w/b ratios, cement types, cement fineness levels, and chemical admixtures. Further studies in this field should explore the possibility of developing a model that

considers both the effect of the w/b ratio and cement type in predicting the rheological properties of cement-based materials.

4.2. Supplementary Cementitious Materials (SCMs)

The rheological behavior of cementitious materials is significantly affected by both the chemical composition and the physical properties of the binder (i.e., mineral and chemical composition, surface area, and granulometry) [27]. For example, Mork and Gjoerv [74] observed a direct correlation between the sulfur trioxide (SO_3) content and the rheological parameters of ordinary Portland cement (OPC) paste, reporting that an increase in the SO_3 content resulted in higher yield stress and plastic viscosity. Conversely, Dils et al. [75] highlighted that OPC pastes with elevated tricalcium aluminate (C_3A) levels and reduced SO_3 content exhibited the most unfavorable rheological properties. In contrast to these findings, García-Maté et al. [76] suggested that the SO_3 content has a negligible impact on the plastic viscosity of calcium sulfoaluminate (CSA) cement paste in its fresh state [76]. On the other hand, Huang et al. [77] found that the structural build-up of cement pastes with a low C_3A content is highly influenced by the variation in the alkali content of the cement. They demonstrated that the addition of alkali in low- C_3A cement paste promotes the electronic interactions between particles via the change in the ionic constitution of the pore solution. These promoted interactions enhanced the agglomeration of cement particles and consequently the structural build-up of cement paste.

Moreover, the influence of binder particle fineness on structural build-up has been investigated. Studies have demonstrated that the rate of structural build-up in cement paste increases with the fineness of the cement particles [78,79]. Finer cement particles provide a larger surface area in contact with water, allowing for faster reactions with water and more effective chemical interactions [80].

However, numerous studies have shown that the changes in rheological properties and the increase in the build-up rate by blending mineral additives are mainly due to the changes in the relative packing density of the cementitious particles, the colloidal interactions, and the cement hydration [81,82]. For example, Huang et al. [27] conducted a detailed investigation into the rheological behavior of blended cements composed of calcium sulfoaluminate clinker (CSA), ordinary Portland clinker (OPC), and anhydrite (CS), with different proportions of OPC, CSA, and CS. Their study revealed that the structural build-up in these OPC-CSA-CS ternary blends is affected by several interconnected mechanisms. These mechanisms include the formation of rigid networks composed of calcium silicate hydrate (CSH), ettringite (AFt), and the gypsum network, along with the contributions of colloidal surface interactions. Additionally, the researchers have shown that the significant role of CSA and CS in accelerating the structural build-up process is attributed to their ability to enhance hydration kinetics. However, it was shown that the w/b ratio, cement type, particle size, and packing density of SCMs appear to play a crucial role in the influence of SCMs on the structuration of cement paste, when contradictory results have been found between different studies on this topic [65]. For example, Baldino et al. [83] reported that limestone powder does not influence the rheological properties of cement paste (slag cement, w/b ratio of 0.45, and 10 wt% of limestone powder with a median diameter of 5 μm). However, Vance et al. [84] reported that the influence of limestone powder on the structural build-up varied depending on several factors, including the type of cement used (binary or ternary blends), the fineness of the limestone (with specific surface areas of 4970 m^2/kg , 2400 m^2/kg , and 613 m^2/kg corresponding to median particle sizes of 0.7 μm , 3 μm , and 15 μm , respectively), the limestone dosage (5, 10, 20, 30, and 40 wt%), and the water-to-binder ratio (0.40 or 0.45). Depending on these parameters, limestone powder was found to either enhance or reduce the structural build-up of the

paste. Similar findings were also reported by Wang et al. [85], who investigated the effects of different types of limestone powder (with specific surface areas of 411, 608, 807, and 1007 m²/kg) used at various dosages (10, 20, and 30 wt%) on Portland cement pastes with a w/b ratio of 0.40. The results showed that incorporating limestone powder at dosages below 10 wt% and with a specific surface area exceeding 600 m²/kg can enhance the structural build-up of fresh cement paste. In contrast, limestone powders with a surface area below 600 m²/kg were found to reduce the build-up. This enhancement observed when the specific surface area exceeded 600 m²/kg is attributed to the finer particle size and higher surface reactivity of the limestone powder, which improve particle packing, provide additional nucleation sites, promote hydration reactions, and contribute to the development of a denser microstructure.

Therefore, the literature shows that the effect of SCMs on the structural build-up of cement-based materials is complex, as it depends on various factors related to the properties of both cement and SCMs. It is also influenced by other parameters, such as the w/b ratio and chemical admixtures. This effect is mainly due to the influence of SCMs on the relative packing density, colloidal interactions, and the cement hydration. With the increasing use of SCMs in cement-based materials to reduce the environmental impact of construction materials, further studies should focus on the various parameters that influence the effect of SCMs on the rheological properties of cement-based materials, such as fineness, shape of particles, and packing fraction. These studies will help in the selection and optimization of SCM integration in sustainable modern concrete.

4.3. Admixtures

The range of organic and mineral additives—commonly referred to as admixtures—used in cementitious materials continues to expand year after year. This growth is due to the need to address diverse construction requirements, enhance the performance, quality, and durability of building materials, and support the development of advanced construction technologies [86–88]. These admixtures play a critical role in improving the workability, viscosity, setting time, strength, and long-term durability of cement-based materials. These admixtures include plasticizers, superplasticizers, viscosity-modifying admixtures (VMAs), air-entraining agents, accelerators, retarders, corrosion inhibitors, and nanomaterials. Due to the vast diversity in their physical and chemical properties, as well as their mechanisms of action, it is challenging to predict or generalize the impact of a specific type of admixture on the structural build-up of cementitious materials. Each admixture interacts differently with cement particles and supplementary cementitious materials depending on its composition, dosage, and mixture, as well as environmental conditions such as cement type, temperature, and humidity. This section provides an overview of and discussion about the effects of the most-used admixtures in cement-based materials, particularly those employed in 3D-printed concrete, such as plasticizers, superplasticizers, viscosity-modifying agents (VMAs), and nanoparticles, on the structural build-up of cement paste. The discussion is based on a review of the scientific literature and considers both the mode of action of these admixtures and the underlying mechanisms contributing to structural build-up.

Plasticizers and superplasticizers such as lignosulphonate, melamine sulphonate, naphthalene sulphonate, and polycarboxylates are incorporated into cementitious materials for their ability to disperse particles effectively. They can adsorb onto the surface of cement particles and modify their surface properties. These agents can either introduce electrical charges (electrostatic repulsion) onto cement particles or attach long polymer chains (steric hindrance) to their surface. As a result, they significantly enhance the repulsive forces, inhibit colloidal interactions, and prevent the flocculation of cement particles [86,89,90]. Additionally, plasticizers and superplasticizers influence cement hydration, mostly by

increasing the dormant period [91]. Consequently, the structural build-up rate of cement pastes decreases with the use of plasticizer and superplasticizer [63,92]. However, due to the limited number of adsorption sites available on the surface of cement particles, the effectiveness of plasticizers and superplasticizers is noticeable only up to a certain dosage (commonly referred to as the saturation point). At this stage, the cement surface becomes fully covered by the admixture molecules, and further additions do not significantly alter the rheological properties of the cement-based material. The exact saturation point depends on both the chemical characteristics of the admixture and the mineralogical composition of the cement [93]. For example, Avina et al. [90] demonstrated that the saturation point corresponds to 0.3% and 2.5% by mass of cement for naphthalene sulfonate and polycarboxylate-based superplasticizers, respectively. This was determined by evaluating the plastic viscosity and static yield stress of cement pastes prepared with Portland cement, a water-to-binder ratio of 0.45, and increasing dosages of superplasticizer. Below the saturation point, both types of admixtures significantly influence the rheological parameters. However, beyond this threshold, no substantial changes in plastic viscosity or static yield stress were observed, indicating that further additions have a negligible effect once the adsorption sites on cement particles are saturated.

VMAs such as clays or nano-clays, welan gum, modified starch, diutan gums, carrageenan, cellulose ethers, alginate, guar gum derivatives, polyethylene oxide, and polyacrylamides are used in cementitious materials to enhance stability by increasing yield stress and viscosity. The action of VMA polymers is associated with several mechanisms, including solvation (interaction between the polar groups of the polymer chains and water molecules, which leads to polymer swelling), swelling (which increases the overall volume fraction of the dispersed phase), entanglement (which contributes to the increase in the viscosity by forming a transient network that restricts the movement of cement particles), and molecular associations (which leads to the reinforcement of the created network through hydrogen bonding or hydrophobic interactions). These interactions lead to the formation of a flocculated three-dimensional network of polymers and cement particles, which increases stress and viscosity. Moreover, attractive interactions between VMA molecules promote the formation of a gel-like structure and inter-particle connections. Consequently, the structural build-up rate of cement pastes increases with the use of VMAs [90,94–98]. Avina et al. [90] studied the impact of various anionic and non-anionic VMAs at different dosages on the rheological behavior of cement pastes. Their findings revealed that the different types and dosages of VMAs can enhance the build-up kinetics of cement paste by improving the internal network and promoting the flocculation of cement particles. In 3D printing applications, the use of VMAs is crucial due to their ability to reduce bleeding, improve shape retention (structural build-up), and delay structural collapse under low stresses [90].

In the last few years, a new class of mineral additives has gained significant attention in cementitious materials. These include nanomaterials such as nano-SiO₂, nano-clay, nano-ZnO, nano-TiO₂, ultrafine phosphorus slag, and nanofibers, which are utilized to enhance the performance of cementitious materials in their fresh or hardened state [99–104]. Nanoparticles characterized by their fitness, high surface energy, and specific surface area influence the packing density of cement particles, as well as the distribution of free water and the flocculation of particles (promote stronger particle–particle interactions and contribute to the formation of a denser network within the paste) in fresh cement paste [105–107]. Additionally, these nanoparticles serve as additional nucleation sites and affect cement hydration [82,108,109]. As a result of these interactions, the incorporation of nanoparticles can lead to an increase in the viscosity and in the structural build-up of fresh cement paste [86]. This effect is dose-dependent; the addition of nanoparticles enhances the structural build-up up to a certain dosage threshold, beyond which a further

increase may lead to a reduction in structural build-up [110]. For example, Jiao et al. [111] investigated the influence of nano-Fe₃O₄ (grain sizes of 20, 30, 100, and 200 nm) with different concentrations (0.25%, 1%, and 3% by mass of cement) on the rheological behavior of cement paste (Portland cement with w/b ratio of 0.40). Their study demonstrated that the structural build-up of the cement paste improved progressively with higher amounts of nano-Fe₃O₄ added. However, Kawashima et al. [112] observed that the structural build-up rate of cement pastes (Portland cement with w/b ratio of 0.43) increased significantly with the use of highly purified attapulgite clays, particularly at 0.5% by mass of cement (rod-like nanoclay with an average length of 1.75 µm and average diameter of 3 nm) during the early stages of hydration. Quanji et al. [110] demonstrated that the addition of a small amount (less than 1.3%) of nano-sized, highly purified magnesium aluminosilicate clay (needle-shaped, with an average diameter of 3 nm and lengths ranging from 1.5 to 2.0 µm) significantly enhanced particle flocculation and the structural build-up of cement pastes (Portland cement with a w/b ratio of 0.4). However, higher dosages were found to reduce the structural build-up. On the other hand, Yuan et al. [113] identified a correlation between the ability of mineral admixtures to accelerate the structural build-up rate and the w/b ratio. They observed that as the w/b ratio decreased, the effect of mineral admixtures on structural build-up was more significant. This trend was consistent across all mineral admixtures tested in their study, which included silica fume, ground slag, fly ash, attapulgite, nano calcium carbonate, and nano silica.

Therefore, mineral admixtures and VMAs increase structural build-up, while plasticizers and superplasticizers have the opposite effect. In cement-based materials, the use of VMAs or mineral admixtures is generally combined with superplasticizers to ensure the desired workability. In 3D printing, buildability, extrudability, and flowability should be ensured in the mix design through the interaction between different admixtures and the effects of other parameters such as the w/b ratio, SCMs, and temperature. Therefore, further studies should focus on the synergistic and antagonistic effects of these parameters to optimize the mix design of modern concrete and minimize its carbon impact by adjusting the dosage of different chemical admixtures.

4.4. Temperature

The structural build-up of cement paste is primarily influenced by the colloidal interactions between cement particles, the nucleation rate, and the quantity of hydration products formed [25]. Variations in temperature can impact the hydration process of cement and the formation of hydration products, thereby affecting the structural build-up kinetics of cement paste. Higher temperatures enhance the solubility of cement particles and accelerate hydration, resulting in increased C-S-H formation (denser) and a faster structural build-up [114–117]. Numerous studies have demonstrated that the impact of temperature on the structural build-up depends on the composition of the cement paste, including the presence of SCMs and mineral or organic admixtures. In all cases, studies on the effect of temperature on the structural build-up of cement paste have shown that its influence is primarily attributed to its role as a hydration accelerator. For SCMs, it was reported that the temperature can affect the pozzolanic reaction rates. Lothenbach et al. [118] showed that the pozzolanic reaction rates of SCMs decreased when the temperature was below 15 °C and increased when the temperature exceeded 27 °C. In line with this finding, Huang et al. [114] studied the influence of temperature on the structural build-up of cement pastes containing various mineral admixtures, including fly ash, slag, and silica fume. Measurements taken at 10, 20, and 40 °C (realistic and frequently encountered environmental conditions in both laboratory settings and field applications) revealed that these admixtures had either no effect or a negative effect on the build-up rate at 10 °C. However, at 20 and 40 °C, positive

effects were observed. The authors attributed these results to temperature-dependent hydration kinetics. At the higher temperatures, the mineral admixtures enhanced the formation and growth rate of C-S-H bridge, whereas this effect was absent at 10 °C. However, Kong et al. [72] demonstrated that higher temperatures enhance the adsorption of polycarboxylate superplasticizers onto cement particles. This increase in adsorption did not result in the improved fluidity of the mixture, nor did it affect the rheological properties.

Therefore, the effect of temperature on structural build-up is primarily linked to its role as a hydration accelerator. However, this effect is strongly influenced by the w/b ratio and other mix properties, such as cement fineness and SCMs [5,93,119,120]. The literature shows that the effect of temperature is straightforward when the w/b ratio is less than 0.5, but becomes more complex and non-linear at higher w/b ratios [121]. In the case of SCMs, the effect of temperature is primarily linked to its influence on pozzolanic reaction rates. Defining a precise threshold temperature is challenging, but according to the literature, a minimum temperature of around 20 °C can enhance the pozzolanic reaction rate and structural build-up.

Cement-based materials undergo temperature changes during preparation, transportation, or placement, and controlling the temperature can be challenging, especially when 3D printing technology is used. Therefore, further studies are needed to better understand the complex interplay between temperature and other parameters and to establish a link between time and temperature variations. This is particularly important, as time also plays a significant role in the effect of temperature on the rheological properties of cement-based materials.

5. Conclusions

This article provides an in-depth overview of the current understanding of structural build-up in cement pastes. It provides a detailed discussion about the measurement methods and influencing factors, such as the w/b ratio, admixtures, temperature, and SCMs. Based on the literature review detailed above, the following conclusions can be drawn:

- The structural build-up is divided into three main stages. These stages are driven by colloidal interactions between cement particles, cement hydration, and inter-particle connection;
- Cement hydration and the formation of hydration products such as ettringite and C-S-H play a crucial role in the evolution of structural build-up. However, further investigation is needed into the effect of the dormant period, during which the dissolution of cement particles supersaturates the pore solution and increases its ionic strength. This condition can affect the structural build-up by promoting particle flocculation and the precipitation of hydrates on the surface of the particles;
- The measurement methods of structural build-up are strongly influenced by the shear rate, shearing time, and disruptive shear technique. The oscillatory shear has a more significant effect on dispersing concentrated cement suspensions than the rotational shear;
- A higher w/b ratio, plasticizers, and superplasticizers decrease the structural build-up, while VMAs, SCMs, mineral admixtures, and temperature have the opposite effect. However, the effect of mineral admixtures is dose dependent. They enhance the structural build-up up to a certain dosage threshold, beyond which a further increase may lead to a reduction in structural build-up;
- The effect of the w/b ratio on structural build-up becomes more complex when SCMs are used, as variations in rheological properties are also influenced by SCM properties such as water demand, morphology, structure, and reactivity;

- The effect of mineral admixtures such as silica fume, ground slag, fly ash, attapulgite, nano calcium carbonate, and nano silica on structural build-up is more pronounced when the w/b ratio decreases;
- The effect of temperature is primarily linked to its role as a hydration accelerator, but it is strongly influenced by the w/b ratio and other mix properties, such as cement fineness and SCMs.

6. Research Needs

In recent years, significant attention has been paid to the time-dependent rheological behavior of cement-based materials, particularly the rate of structural build-up, which is influenced by early-age physical interactions between particles and cement hydration processes [23,94,121]. The structural build-up of concrete plays a critical role in its performance across various applications, including cast-in-place concrete, 3D printing, and slip-form paving construction. In cast-in-place concrete, the rate of structuration build-up is particularly important, because it directly impacts the maximum lateral pressure exerted on formwork, as well as its gradual reduction over time. A higher build-up rate leads to reduced lateral pressure, which helps to lower formwork costs and enables higher placement heights [122]. In 3D printing, a faster build-up rate ensures that the material can support subsequent layers and form strong bonds between them [123–125]. Similarly, in fluid concrete with adapted rheology, a faster build-up rate enhances resistance to segregation and bleeding, leading to better interface quality between aggregates and cement paste [126]. These factors collectively improve the durability of cementitious materials by reducing permeability, enhancing adhesion to reinforcements, and boosting mechanical performance [62,127]. However, while this structural build-up process improves durability, it can also reduce fluidity, posing challenges for construction activities that require high workability, such as transport, pumping, and placement [128,129]. Therefore, controlling the rate of structural build-up in the early stages is essential for achieving a balance between durability and workability, ensuring the production of high-quality concrete structures. This task, however, is complex, due to the numerous influencing factors and their interactions with the cement hydration process. While this review has highlighted the effects of various individual factors, the interdependencies among them create significant challenges in predicting and controlling the structural build-up rate across diverse construction scenarios. For instance, the impact of temperature on hydration kinetics can be either amplified or moderated depending on the presence of specific admixtures. Similarly, the influence of SCMs on the build-up rate is impacted based on the w/b ratio, as well as the physical and chemical properties of both the cement and the SCMs. Additionally, the growing use of non-conventional materials, such as ultra-fine mineral admixtures, adds another layer of complexity to predicting the rheological properties and structural build-up of cement paste [130]. To address these challenges, advanced rheological models and real-time monitoring techniques have become indispensable. These tools are essential for capturing the dynamic behavior of cement pastes, accounting for the interactions between the temperature, admixtures, SCMs, and other variables. By enabling more accurate predictions and better control of the structural build-up rate, these approaches contribute to the development of cement-based materials tailored to meet the specific demands of various construction applications.

Ultimately, integrating advanced modeling and monitoring technologies into the design process will not only improve the balance between workability and structural build-up, but also enhance the overall efficiency, durability, and sustainability of modern construction practices. These innovations are vital for addressing the evolving challenges

in the construction industry and ensuring the continued advancement of high-performance cementitious materials.

Author Contributions: M.H. and Y.E.B. conceptualized the review structure and led the writing, including original draft preparation and editing. K.B. and A.Y. supervised the project, provided critical revisions, and helped shape the manuscript's direction. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest.

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